NASA-CR-195789

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26 April, 1994

MEMO TO: Mrs. Sonia M. Schriver, Grants Officer, MS 500-313, NASA Lewis Research Center, Cleveland, OH.

FROM: Dr. Mehmet Uz, Associate Professor. Mehmet Uz

SUBJECT: Final Report for Cooperative Agreement NCC3-267.

Combining a one-semester sabbatical from Lafayette College with a research grant from NASA (Cooperative Agreement NCC3-267), I worked in Collaboration with Robert H. Titran, Advanced Metallics Branch, Materials Division at NASA Lewis Research Center in Cleveland, Ohio, from 1 June 1992 through 31 July 1993. Attached is a brief summary of my work which involved processing, microstructure and properties of Nb-1Zr-C alloys in both the sheet and tube forms, and also the publications and presentations that resulted. I have also enclosed the copies of the NASA reports some of which are published in the open literature as indicated.

Please contact me at (610) 250-5408 by phone or at (610) 250-5059 by fax should you need any further information.

(NASA-CR-195789) FINAL REPORT (Lafayette Coll.) 5 p

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Attachments
Enclosures

cc: Dr. Robert V. Miner, Chief, Advanced Metallics Branch, Materials Division, NASA Lewis Research Center, Cleveland, OH.

NASA Center for Aerospace Information, BWI Airport, MD.

Lafayette College Research Office, Easton, PA.

Dr. Mehmet Uz, Final Report, Cooperative Agreement NCC3-267

WORK ON SHEET

Sheets of Nb-1Zr-0.06C and Nb-1Zr-0.1C were evaluated to investigate the effects processing and carbon content on the microstructure and mechanical properties including long-term (10,000 to over 34,000 hours) creep behavior, tensile properties and microhardness. Three different Nb-1Zr-0.1C sheets fabricated by cold rolling following single, double or triple extrusion operations at 1900 K were examined to asses the effects of thermomechanical processing.

All the high temperature tests were performed in ultrahigh vacuum chambers. The samples were given a double-anneal heat treatment (DA: 1 h @ 1755 K and 2 h @ 1475 K) prior to creep or tensile testing. The creep tests were carried out at 1350 K under both 10 and 34.5 MPa for times ranging from a few hundred to over 34,000 hours depending on the strains developed during testing. Tensile tests were made at both 300 and 1350 K using a screw-driven testing machine. Microstructures of the samples were characterized before and after any heat treatment and creep testing to determine the effects of processing on the microstructure and its long term stability at elevated temperatures. A combination of metallographic techniques were used to characterize the microstructure including grain size, and the crystal structure and morphology of the precipitates.

The results of the studies on the processing, microstructure and properties of Nb-Zr-C alloy sheets can be summarized as follows:

1. The tensile properties of the Nb-1Zr alloys with and without 0.1C are comparable to one another at room temperature as well as at 1350 K. The number of hot extrusions performed prior to cold rolling does not appear to have any significant effect on the tensile properties of Nb-1Zr-0.1C sheets at 300 or 1350 K.

2. The microhardness of the single-, double- and triple-extruded Nb-1Zr-0.1C sheets were comparable to one another in a given condition, namely cold-rolled, double-annealed and crept. The microhardness of any of the samples was not affected by exposure to 1350 K for as long as 19,000 hours following the double-anneal heat treatment.

3. The creep resistance of each of the carbide-strengthened Nb-1Zr-C sheets was much superior to that of solid solution-strengthened Nb-1Zr at 1350 K under either 10 or 34.5 MPa. Furthermore, the creep resistance of the alloy containing 0.1C was much better than that with 0.06C.

4. The number of extrusions did not seem to affect the long term stability of the microstructure and the creep behavior of the Nb-1Zr-0.1C sheets at 1350 K and 34.5 MPa.

5. The only precipitates detected in the Nb-1Zr samples were fine oxides of ZrO_2. The precipitates were primarily rather coarse carbides of orthorhombic Nb_2C in all of the cast, extruded and cold-rolled samples containing carbon. However, these carbides were found to transform to finer and extremely stable cubic carbides of (Zr,Nb)C upon heat treatment and/or during the subsequent prolonged exposure to 1350 K. The only carbides in the stressed middle and the stress-free ends of each of the crept Nb-1Zr-C sheets were finely-distributed cubic (Zr,Nb)C which provide these alloys with the excellent high temperature microstructural stability and creep resistance.

WORK ON TUBES

Microstructure of Nb-1Zr-0.1C tubes were characterized as affected by extrusion temperature of the tube shell and its thermomechanical processing to tubing. Two tube shells of about 40-mm outside diameter (OD) and 25-mm inside diameter (ID) were extruded 8:1 from a vacuum arc-melted ingot at 1900 and 1550 K. Two different OD tubes of ~0.36-mm wall thickness were fabricated from each tube shell by a series of 26 cold drawing operations with two in-process anneals. The microstructure of tube shells and the tubing before and after a 2-step heat treatment were characterized. Residue extracted chemically from each sample was also analyzed to identify the precipitates. The results showed that an 8:1 extrusion at 1900 K yielded a tube shell with a finer microstructure than the same at 1550 K. Both extrusions resulted in break-down of the as-cast grain structure and some transformation of Nb_2C to the stable cubic carbide (Zr,Nb)C. The as-drawn and double-annealed tubes from either extrusion were similar in both microstructure and microhardness indicating that the effect of the extrusion temperature on the tube shells was not transmitted to the final tubing from them.

The results of this study on tubes is consistent with those we obtained on sheets in that thermomechanical processing resulted in refinement of the microstructure and transformation of Nb_2C to extremely stable (Zr,Nb)C. The microstructure of all the tubing resembles that of a Nb-1Zr-0.1C sheet (with a much different processing history than the tubes) which we showed to have excellent high temperature stability and creep resistance. This would indicate that materials with similar microstructure and properties can be obtained by different processing schedules.

Based on our experience with sheets and the results of this work, we conclude that the subsequent processing rather than the initial extrusion conditions dictated the properties of the final Nb-1Zr-0.1C sheet and tubing. However, more mechanical property testing of the tubes is needed to confirm the premise that the tubes have similar properties to the alloy sheet.

Dr. Mehmet Uz, **Final Report, Cooperative Agreement NCC3-267**

PUBLICATIONS AND PRESENTATIONS THAT RESULTED FROM RESEARCH AT NASA LEWIS RESEARCH CENTER

Mehmet Uz and R. H. Titran. **"Processing and Microstructure of Nb-1%Zr-0.1%C Alloy Sheet."**

Published as:

NASA TM - 105921. NASA LeRC, Cleveland, Ohio, 1993, and in

AIP (American Institute of Physics) Conference Proceedings 271, DOE CONF-930103, Mohamed S. El-Genk and Mark D. Hoover, eds., New York, NY, 1993, pp. 69-83.

Presented at:

Materials Division Seminar, NASA Lewis Research Center, Cleveland, OH, 6 January 1993, and

Tenth Symposium on Space Nuclear Power and Propulsion, Session [2]: Fuels and Materials, Albuquerque, NM, Jan 10-14 1993.

Mehmet Uz and Robert H. Titran. **"Effects of Processing and Prolonged High Temperature Exposure on the Microstructure of Nb-1%Zr-0.1%C Sheet".**

Published as:

DOE/NASA/16310-20, NASA TM-106370, NASA LeRC, Cleveland, OH, 1993. and in

High Temperature Silicides and Refractory Alloys, C. L. Briant, J. J. Petrovic, B. P. Bewlay, A. K. Vasudevan and H. A. Lipsitt, eds., MRS Symposium Proceeding Series, Volume 322, Pittsburgh, PA, 1994, pp. 443-452.

Presented at:

Symposium F: High Temperature Silicides and Refractory Alloys, held during 1993 MRS Fall Meeting, Boston, MA, 29 November - 2 December 1993.

Mehmet Uz and Robert H. Titran. **"Characterization of the Microstructure of Nb-1 wt.%Zr-0.1 wt.%C Tubes as Affected by Thermomechanical Processing".**

Published as:

DOE/NASA 16310-21, and NASA TM -106381. NASA LeRC, Cleveland, OH, December 1993. and in

Dr. Mehmet Uz, Final Report, Cooperative Agreement NCC3-267

AIP Conference Proceedings 301, Mohamed S. El-Genk and Mark D. Hoover, eds., New York, NY, 1994, pp. 393-402.

Presented at

Eleventh Symposium on Space Nuclear Power and Propulsion, Albuquerque, NM, 9-13 January 1994. (Presented by my co-author Mr. Titran).

Robert H. Titran and Mehmet Uz. **"Effects of Thermomechanical Processing on the Microstructure and Mechanical Properties of Nb-Zr-C Alloys".**

Abstract published in the Program of the Specialist Conference on Space Nuclear Power and Propulsion Technologies - Materials and Fuels (NEMF-93), Podolsk, Moscow Region, Russia, 21-24 September 1993, and the paper is presented at the same conference.

Robert H. Titran and Mehmet Uz. **"Effects of Thermomechanical Processing on Tensile and Long-Time Creep Behavior of Nb-1%Zr-0.1%C Sheet".**

Published as:

DOE/NASA 26310-19, and NASA TM -106319. NASA LeRC, Cleveland, OH, December 1993.

Presented at:

The **Deformation Session** during the 123rd TMS Annual Meeting, San Francisco, CA, 27 February - 3 March 1994.